

PROCESSING AND CHARACTERIZATION OF CARBON NANOTUBE COMPOSITES*

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ABSTRACT

Recent advances in the synthesis of large-scale quantities of carbon nanotubes (CNT) have provided the opportunity to study the mechanical properties of polymer matrix composites using these novel materials as reinforcement. Nanocomp Technologies, Inc. currently supplies large sheets with dimensions up to 122 cm x 244 cm containing both single-wall and few-wall CNTs. The tubes are approximately 1 mm in length with diameters ranging from 8 to 12 nm. In the present study being conducted at NASA Langley Research Center (LaRC), single and multiple layers of CNT sheets were infused or coated with various polymer solutions that included commercial toughened-epoxies and bismaleimides, as well as a LaRC developed polyimide. The resulting CNT composites were tested in tension using a modified version of ASTM D882-12 to determine their strength and modulus values. The effects of solvent treatment and mechanical elongation/alignment of the CNT sheets on the tensile performance of the composite were determined. Thin composites (around 50 wt% CNT) fabricated from acetone condensed and elongated CNT sheets with either a BMI or polyimide resin solution exhibited specific tensile moduli approaching that of toughened epoxy/ IM7 carbon fiber unidirectional composites.

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1.0 INTRODUCTION

A material configuration long touted as being a theoretically promising advancement in material development utilizes nano-sized particles in the form of sheets of graphene rolled into long, slender tubes, commonly referred to as carbon nanotubes (CNTs). These nanotubes may exist as a single sheet of rolled graphene referred to as single-walled carbon nanotubes (SWNTs), or as a system of concentric nanotubes referred to as multi-walled carbon nanotubes (MWNTs). SWNTs are characterized as having diameters of 1-10 nm and varying lengths of up to a million times their diameter [1].

A single carbon fiber filament, which is the basis for the most advanced polymeric composite materials commercially in use today, has a diameter of 5-10 μm [2]. When tested in a tow, or bundle, of 6000 filaments, it has a tensile strength of 5.67 GPa and a tensile modulus of 0.276 TPa [2]. Individual SWNTs have been tested and have a tensile strength of 13-53 GPa and a tensile modulus of 1-5 TPa [3-5]. Compared with carbon fibers, the strength to weight ratio of nanotubes in the axial direction is up to four times greater [6]. It is these extremely high properties, which have been demonstrated through experimental testing on the nanoscale, that have led to a significant amount of research [7-12] into utilizing these nanometer-sized particles as the reinforcement for the next generation of advanced composite materials. Typical mechanical data for various aerospace materials are plotted in Figure 1. It is evident from the figure that achieving only a portion of the nanoscale properties of CNT's in a macroscale composite could offer a significant potential improvement compared to current materials.

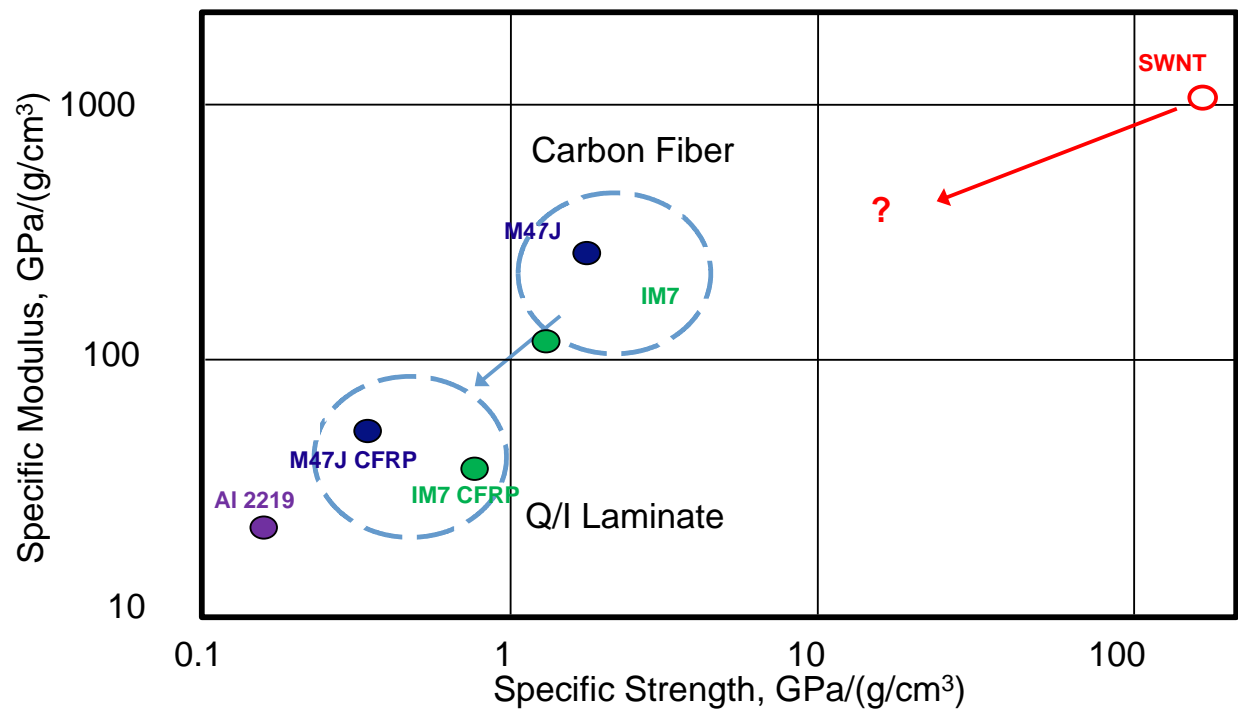


Figure 1. Mechanical data for materials commonly used in aerospace applications [Harris, Shuart, Gray, NASA TM 211664, 2002].

While the mechanical performance of CNTs has significant potential, the main challenge which the technology has faced since gaining significant interest in 1991 is the ability to translate these nanoscale properties to the macroscopic level to be utilized in aerospace structures. These challenges stem from the tendency of CNTs to bundle together and entangle, forming agglomerates, which leads to defect sites within the composites and limits the efficiency of CNTs as reinforcements in polymer matrices [13]. Numerous studies have been conducted to determine the effect of the dispersion and alignment of CNTs on the mechanical properties of CNT/polymer composites. It has been observed by numerous studies [14-16] that poor dispersion and alignment of the CNTs leads to a reduction of properties, such as fracture toughness and tensile strength, compared to the pristine resin. The dispersion of CNTs for the purpose of doping matrix resins with CNT reinforcements has not proven to be an effective means of incorporating sufficient CNT content to yield mechanical properties that are competitive with carbon fiber reinforced polymeric (CFRP) composites typically used in aerospace structures.

Researchers have tried numerous techniques to increase the alignment of CNTs in polymeric composites. Many of these methods are 'in-situ' and involve dispersing and aligning freestanding CNTs incorporated into the polymer matrix. These methods include dielectrophoretic force field-induced alignment [17-19], magnetic field-induced alignment [20,21], electrospinning-induced alignment [22-25], and liquid crystalline phase-induced alignment [26-29]. Conversely, many 'ex-situ' alignment techniques where the CNTs are aligned in advance and then combined with a polymer matrix have been explored as well. Methods utilizing filtration [30-32], plasma-enhanced chemical vapor deposition [33,34], and mechanical stretching [35,36] have all been examined, with little success in achieving the level of macroscale mechanical property enhancements necessary to significantly advance the state of the art in structural composites.

Recent advances in the synthesis of large-scale quantities of carbon nanotubes (CNT) in formats amenable to structural composite processing have provided the opportunity to study the mechanical properties of polymer matrix composites using these novel materials as reinforcement. Nanocomp Technologies, Inc. currently supplies large sheets with dimensions up to 122 cm x 244 cm containing both single-wall and few-wall CNT. The tubes are approximately 1 mm in length with diameters ranging from 8 to 12 nm. The manufacturing process for their material is considered a 'dry' method since there is no need for the CNTs to be placed in a suspension as is typical for the fabrication of conventional bucky papers [37,38]. Instead, the CNTs are synthesized with a CVD process and directly deposited onto a moving belt in a semi-continuous process [39]. The schematic for the patented process developed by Nanocomp is shown in Figure 2.

The nanotubes are intrinsically aligned along the rolling direction of the belt. When tensile tested along this direction and perpendicular to it, there are apparent differences in the mechanical properties [39]. SEM images of typical CNT sheets are shown in Figures 3 and 4. A further solvent treatment is also used by Nanocomp to condense and improve the performance of the material.

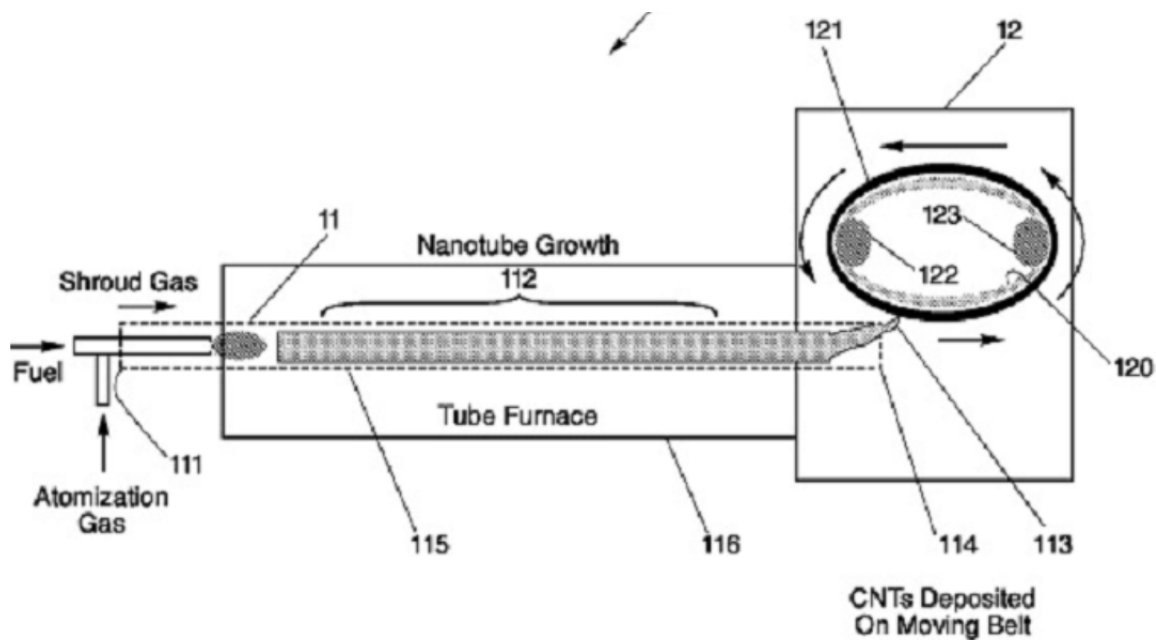


Figure 2. Schematic of Nanocomp Technologies, Inc. CNT sheet production method. (U.S. Patent 8246886) [39]

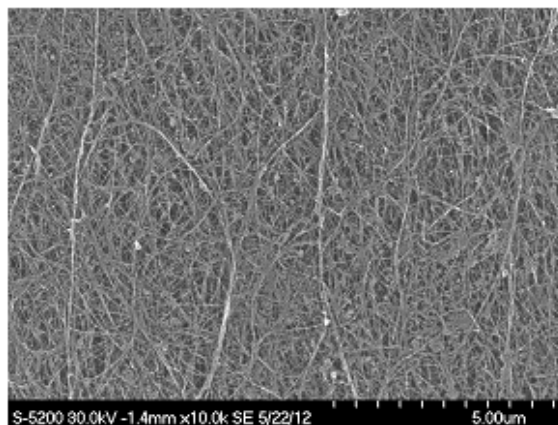


Figure 3. Image from High Resolution SEM of CNT sheet surface, 10 μm x 10 μm area.

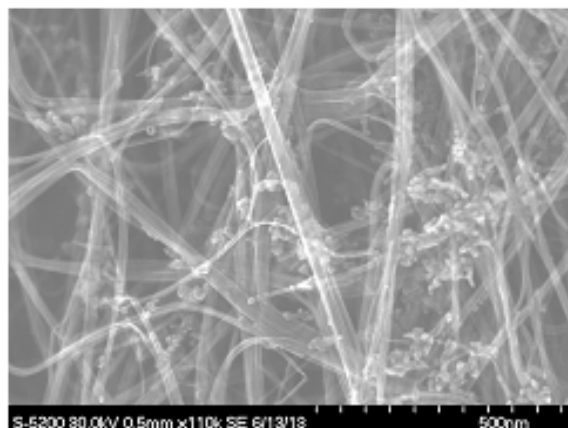


Figure 4. Image from High Resolution SEM of CNT sheet surface, 1 μm x 1 μm area.

In order to further the state-of-the-art in lightweight CNT composite materials and increase their potential use as a structural material, CNT sheets obtained from Nanocomp, Inc. were processed into composites with various resins and evaluated for tensile mechanical properties. The focus of the research presented here is to evaluate and improve the mechanical properties of CNT-based polymer matrix composites by increasing the alignment of the CNTs within the sheets using processes that can be scaled up to produce larger composite articles.

2.0 EXPERIMENTAL

2.1 Materials

Four resin systems were evaluated in this work: RM-3010, a bismaleimide (BMI) obtained from Renegade Materials Corporation, Miamisburg, OH, USA with a tensile strength of 124 MPa and an elastic modulus of 4.59 GPa. RM-3010 was used as a 5 wt% solution of BMI in methylethylketone (MEK) or dimethylacetamide (DMAc). LaRC PETI-9, a polyimide developed at NASA Langley, obtained from Imitec, Inc., Schenectady, NY, USA was used as a 15 wt% solution imide in N-methyl-2-pyrrolidone (NMP). Specific details on the processing and chemistry of LaRC PETI-9 are available in Reference 40. API-60, a toughened epoxy obtained from Applied Poleramic, Inc., Benicia, CA, USA with properties similar to Hexcel 8552 (tensile strength of 121 MPa and an elastic modulus of 4.7 GPa). API-60 was used as a 5 wt% solution of epoxy in MEK/NMP (50/50). Ultem 1000, a polyetherimide (PEI) obtained from GE Plastics, Schenectady, NY, USA with a tensile strength of 105 MPa and an elastic modulus of 2.9 GPa. Ultem 1000 was used as a 12.5 wt% solution of PEI in NMP.

Acetone condensed CNT sheets, Lot #5932 and Lot #5768 (122 cm x 244 cm), were obtained from Nanocomp Technologies, Inc., Merrimack, NH.

2.2 CNT Sheet Stretching/Alignment

In order to increase the alignment of the CNTs, CNT sheets were stretched to approximately 121% of their original length (10.8 cm in. to 13.1 cm) with the in-housed developed and fabricated stretching apparatus shown in Figure 5. CNT sheets were cut into 12.7 cm by 20 cm or 12.7 cm by 23.5 cm specimens and stretched to align CNTs in CNT sheets following the procedure shown in Figure 6. Some specimens were stretched, removed from the apparatus and then cut to 7.6 x 7.6 cm specimens for composite fabrication. Some specimens were stretched, clamped and held in tension with a tool and then processed into composite laminates as shown in Figures 7 and 8. Larger starting specimens were required for the clamping tool. As shown in Figure 7, some samples were coated with resin post stretching while other samples were coated with resin and then stretched, clamped and held in tension as shown in Figure 8. Specimens stretched without resin will be referred to as 'dry' while specimens stretched after being painted/coated with resin solution will be referred to as 'wet'. All CNT composite panels described in Section 2.3 were fabricated from the center of the stretched area to minimize variations in stretching that occurred at the edges of the starting larger specimens.

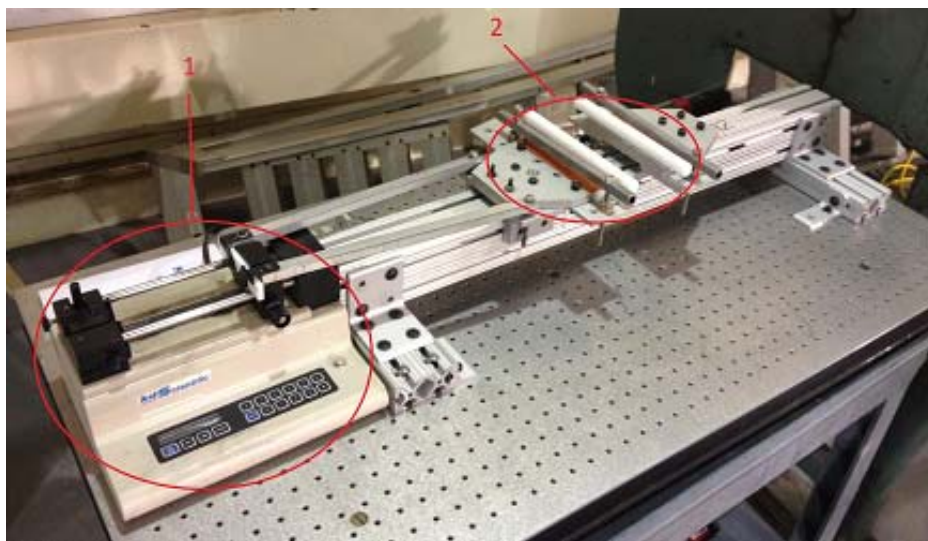


Figure 5. Mechanical stretching apparatus designed to stretch CNT sheets. 1: KD Scientific Model 410 syringe pump. 2: Clamps designed to hold the CNT sheets.

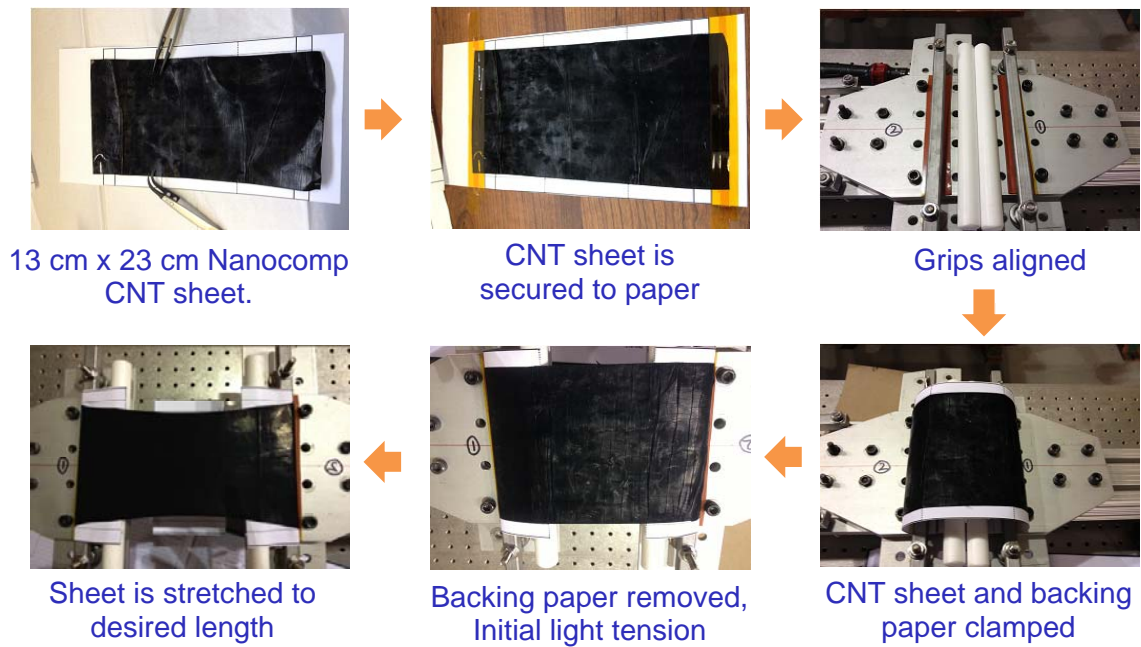


Figure 6. Procedure to stretch CNT sheets.

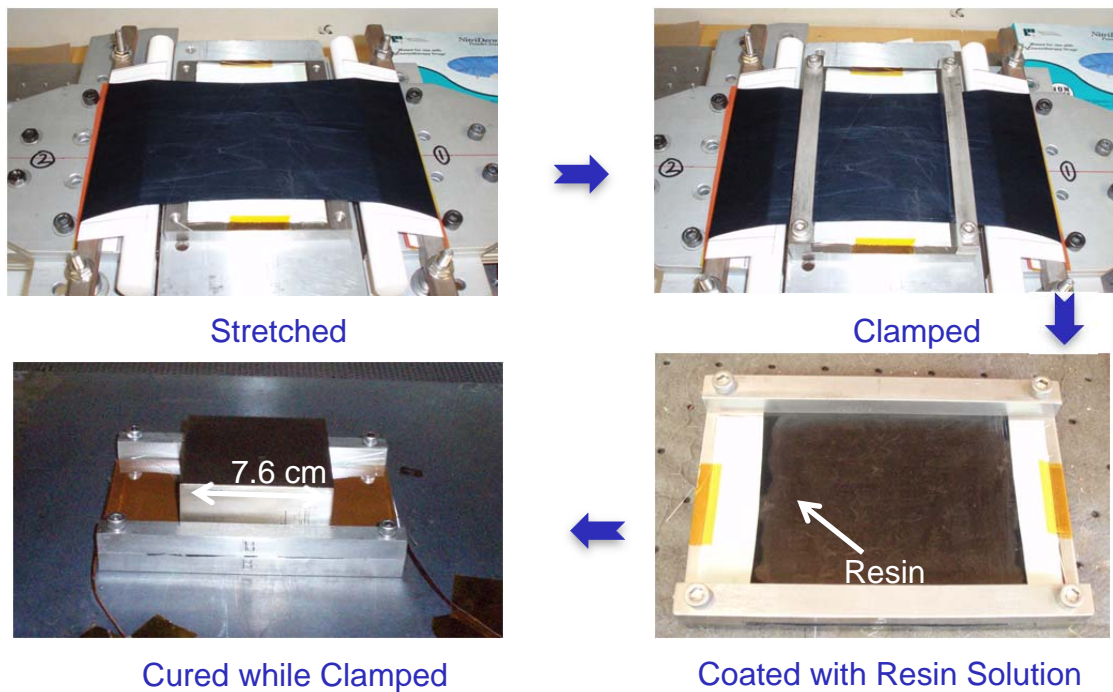


Figure 7. CNT sheet clamping procedure with 'dry' CNT sheets.

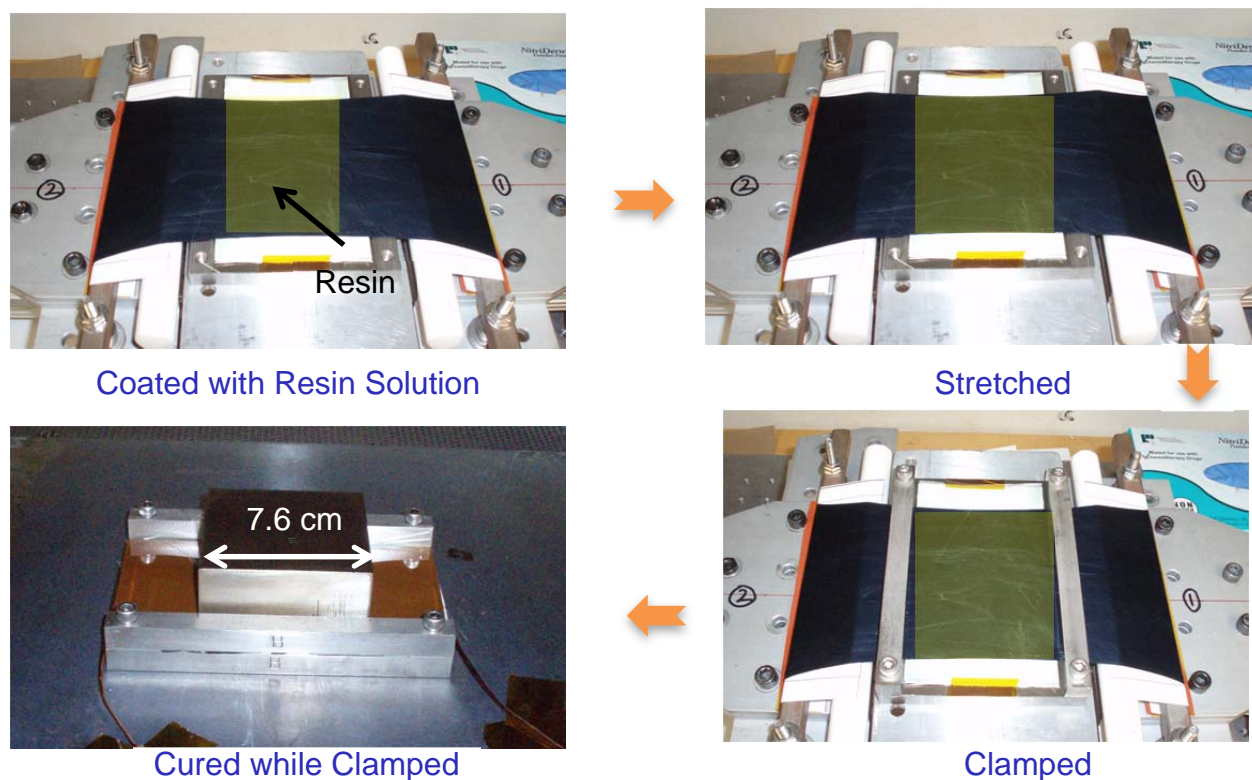


Figure 8. CNT sheet clamping procedure with 'wet' CNT sheets.

2.3 Composite Fabrication

All CNT composites were fabricated by coating CNT sheets with solutions of the resins described in Section 2.1 by hand using a brush to achieve a nominal 50wt% CNT/resin distribution. The appropriate amount of solution was measured prior to application and applied as evenly as manually possible onto the CNT sheets. CNT composites were all cured using a stainless steel mold or the mold shown in Figures 7 and 8 in a vacuum press utilizing the recommended cure cycle for each resin system. Resultant CNT weight fractions are shown in Table 1. Weight fractions were determined using after-cure composite weight and average CNT sheet areal density.

2.4 Composite Mechanical Properties

Tensile mechanical properties were measured using a MTS 858 test stand following the ASTM D-638 standard for plastics testing. The testing was performed using the specimen geometry shown in Figure 9. A LX 300 laser extensometer was utilized to determine strain from the CNT composites.

Table 1. CNT composite characteristics.

Test Coupon CNT Weight Fractions, wt%							
Material	Baseline 2-ply	Stretched 2-Ply	Stretched/ Held 1-ply	Stretched/ Held 2-ply	Stretched/ Held 1-ply Repeat	Stretched/ Held 1-ply Repeat-2	Coated-> Stretched/ Held 1-ply
Epoxy: API-60	66	54	53	61	65	n/a	65
Polyimide: LaRC PETI-9	56	47.8	43	53	54	n/a	47
BMI: RM-3010	52	52	50	53	51	54	75
PEI: Ultem 1000	53	55	58	n/a	n/a	n/a	n/a

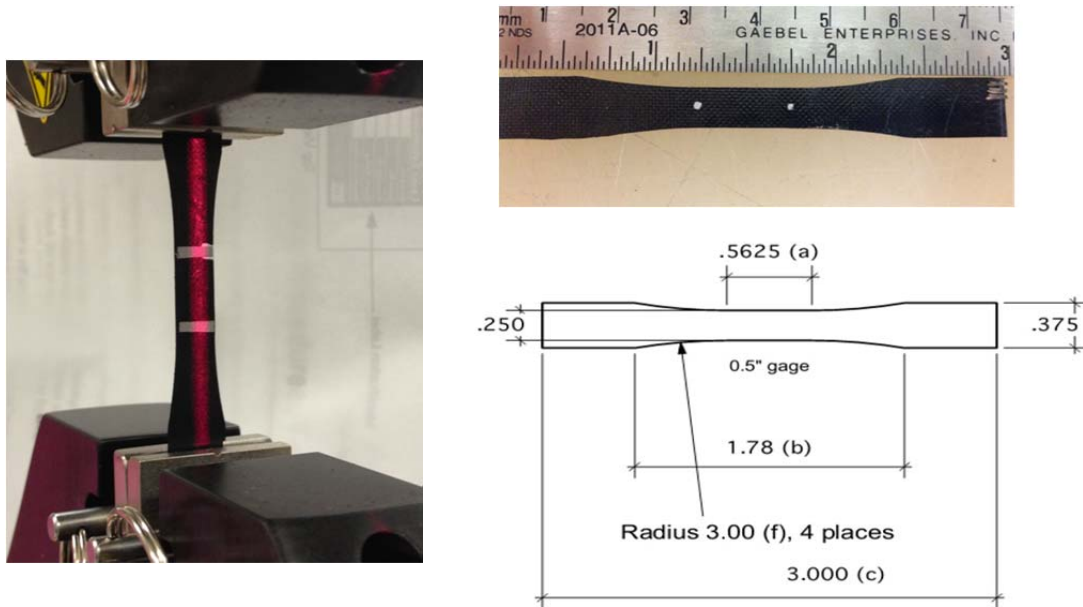


Figure 9. CNT composite tensile test coupon configuration. **Left:** CNT specimen in test grips with laser extensometer **Top:** CNT specimen. **Bottom:** Schematic of CNT tensile specimen with dimensions in inches (1 in equals 2.54 cm).

3.0 RESULTS AND DISCUSSION

3.1 Mechanical Test Development

A reliable method for measuring tensile strength and stiffness of scaled-up CNT composites was developed as shown in Figure 9. Previous testing efforts suffered from the difficulty of

measuring strain on small, delicate specimens and a proper definition of the region where to measure stiffness. The utilization of the ASTM D-638 standard, along with a LX 300 Laser Extensometer to measure strain directly from the CNT composite specimens produces more consistent and reliable data. A typical stress strain curve for CNT composites obtained by the testing configuration developed and utilized in this work is shown in Figure 10. The location on the curve used to report modulus was determined by evaluating the variation of modulus from a series of tests over a range of loads. It was determined that the most consistent data was obtained at the 10 to 30% of max load range. Modulus data in this range resulted in lower standard deviations than other regions in the 5 to 50% of load range, which was determined to be the overall region of interest to determine modulus. Therefore, all of the modulus data reported in Section 3.2 were obtained from the slope of the stress/strain curve from laser extensometer data over a range of 10 to 30% of maximum load. The red portion of the curve shown in Figure 10 illustrates the part of the curve typically used to determine modulus. A more detailed explanation of this work can be found in Reference 41.

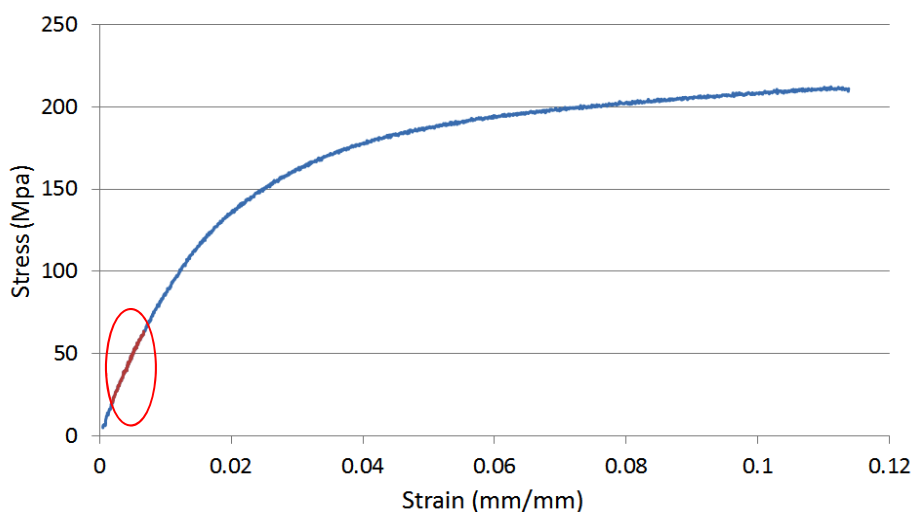


Figure 10. Typical stress/strain curve for 2-ply CNT composite.

3.2 Mechanical Properties

Mechanical properties of various CNT composites are presented in Figures 11 and 12. Data are presented for baseline CNT only sheets, 2-ply CNT composites without stretch, 2-ply stretched CNT composites, 1-ply CNT composites from stretched and clamped/held 'dry' sheets, 2-ply CNT composites from stretched and clamped/held 'dry' sheets and 1-ply CNT composites from stretched and clamped/ held 'wet' sheets. As shown in Figure 11, the addition of resin to form a composite increased the tensile strength of the baseline sheet for each resin type evaluated. For the epoxy and BMI resins, stretching the CNT sheets further improved the tensile strength. However, clamping the stretched CNT sheets to eliminate any relaxation did not significantly alter the tensile strength. For the polyimide and PEI resins, stretching the CNT sheets did not

significantly affect the tensile strengths within the scatter of the data. The 2-ply stretched and clamped/held in tension composite made from PETI-9 demonstrated a significant improvement in tensile strength. The difference observed between a 1-ply and 2-ply composites made by the same procedure could be a result of variations in the starting material, as well as differences in the CNT weight fractions shown in Table 1. The 1-ply stretched and clamped data was repeated to verify reproducibility of the data. Repeat data of this panel configuration resulted in slightly higher tensile strength, though still within the scatter of the data. A second repeat experiment of the BMI data likewise demonstrated a slight increase in tensile strength. Some of this improvement may be attributed to improved specimen preparation with experience handling these CNT sheets. Variation in the CNT sheet and CNT weight fraction may also explain some of the differences seen in repeat experiments. Stretching the CNT sheets while ‘wet’ with resin solution and curing while held in tension/clamped did not yield significant improvements in tensile properties. The epoxy and BMI data were similar to repeat ‘dry’ data and the polyimide sample was similar to the 2-ply stretched and clamped data. Overall, modifications to the CNT sheets and the composite processing techniques evaluated in this work resulted in CNT composites with tensile specific strengths of around 550 MPa/(g/cc) that are significantly lower than current carbon fiber composites.

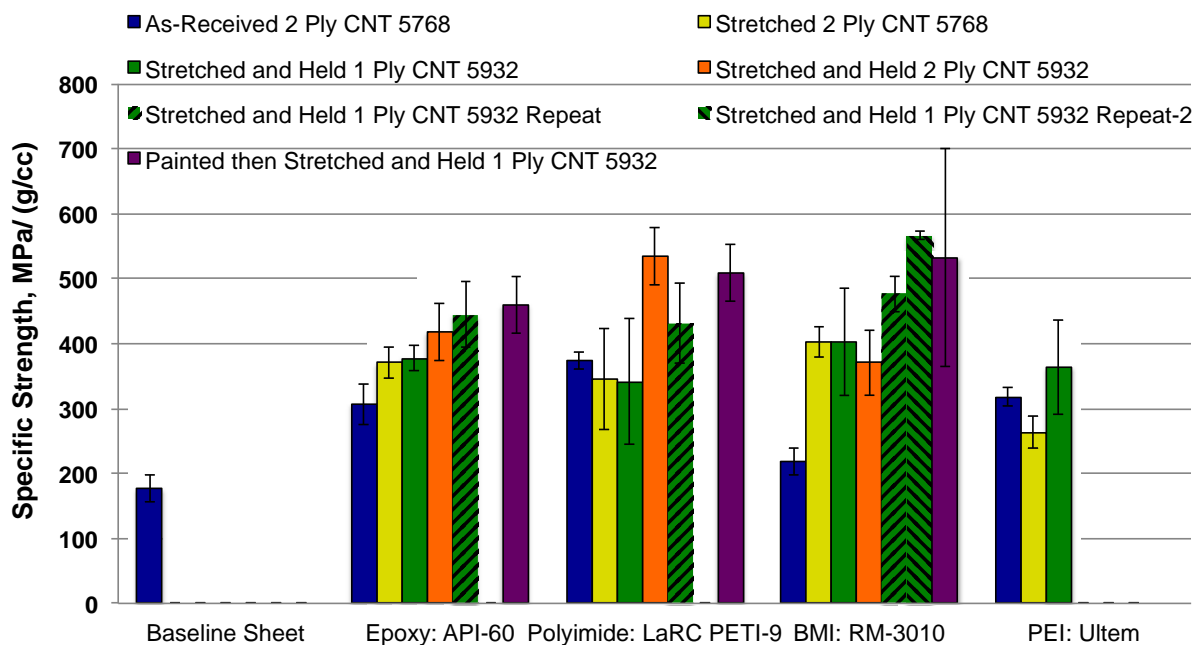


Figure 11. Tensile specific strength of acetone treated CNT composites.

A more significant improvement in tensile modulus was evident from the introduction of resin and a further increase from the stretching process. Baseline as-received sheets demonstrate extremely low modulus values that are barely visible in the scale used in Figure 12. CNT composites that were not stretched had much higher modulus values. Stretching alone further improved the modulus values for all four resin types. One-ply composites that were stretched

‘dry’ and held in tension during cure further improved modulus values. Both the polyimide and BMI composites demonstrated specific moduli of over 80 GPa/(g/cc) with individual specimens having values over 100 GPa/(g/cc). These 1-ply composite data are very promising as the specific modulus of IM7/ 8552 unidirectional composites was 104 GPa/(g/cc). However, the 2-ply composites using stretched ‘dry’ and clamped CNT sheets resulted in much lower values. A repeat of the 1-ply stretched and clamped data were less than the initial modulus values. However, the polyimide composite repeat data was well above 60 GPa/(g/cc). A second repeat of the BMI 1-ply data did not duplicate the initial high modulus values. The composite modulus data from the stretched ‘wet’ CNT sheets did not achieve the highest values obtained in other configurations. It should be noted that due to the volatility of MEK, a 5% solution of BMI in DMAc was utilized for the BMI ‘wet’ stretching procedure.

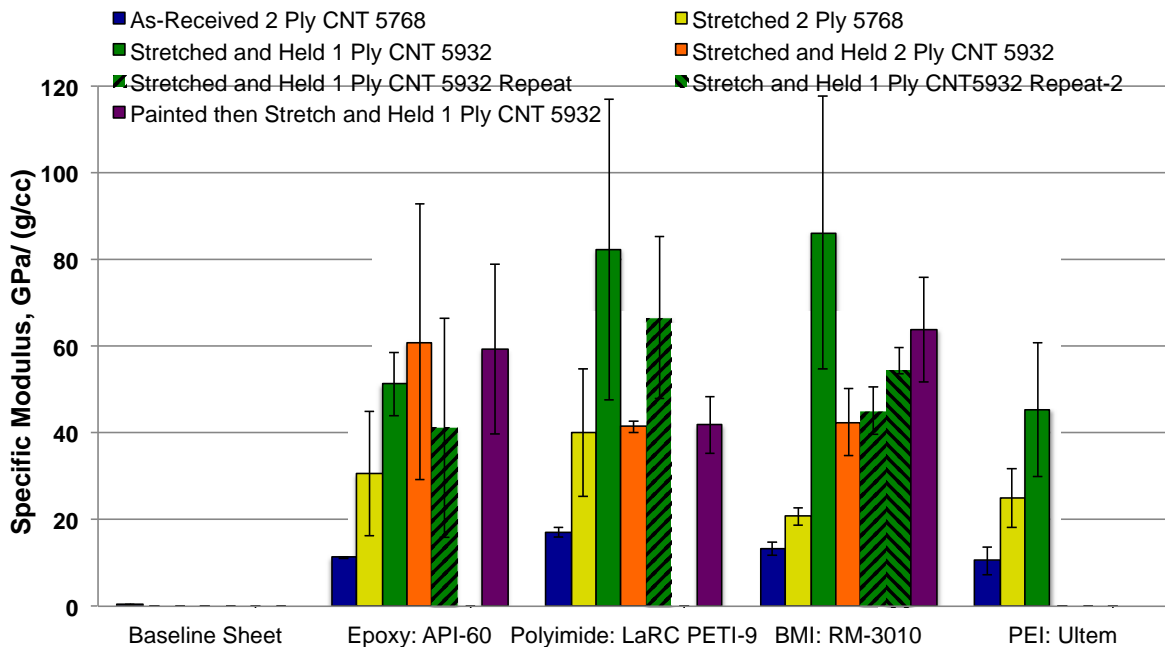


Figure 12. Tensile specific modulus of acetone treated CNT composites.

4.0 CONCLUSIONS

Methods for mechanical stretching, fabrication, and mechanical testing have been developed for characterizing CNT composites fabricated from Nanocomp’s CNT sheets. Stretching provides an improvement in strength and modulus compared to the baseline properties. A greater improvement in properties was evident in the modulus values of BMI and PETI-9 composites. Significant standard deviations are evident in the data and reproducibility of the results was deficient. At this point in the effort, the CNT composite properties are not yet equivalent to

standard carbon composite properties. The results suggest that further modifications to the CNTs, along with further process optimization are required to potentially improve mechanical performance at the macro scale and ultimately surpass those of carbon fiber composites.

5.0 FUTURE WORK

Future work will include the evaluation of various functionalization techniques to improve composite panel properties. Exploration of alternative processing methodologies to improve and optimize the control of resin content and wetting of CNTs will be carried out. Alternative CNT starting material formats such as yarns and CNT arrays will be evaluated.

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